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of Nitric Acid Treatment of Nickel Wire Mesh

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SUPPLEMENT TO REPORT NO. 25X1X

When very fine ("finest") nickel wire mesh is treated with 20% nitric acid at 55° over a period of 3 minutes, it turns out that when the mesh is submerged vertically certain warp wires are dissolved out of what appears to be still intact mesh. Repeated experiments show the same results. A series of investigations was undertaken to clear up this phenomenon.

The first thing to be determined is the 30% loss in weight after the above-mentioned treatment takes place. It is further evident that certain individual wires are not completely dissolved, but that some small remnants of them remain. Figure 1 is a microphotograph of a piece of untreated mesh. Figure 2 shows a section of the same mesh after the nitric acid test. As can be seen, certain individual warp wires are completely dissolved out, 3 warp wires are to a certain extent completely (sic) dissolved out, and to a certain extent they are still there but in a very thin form. One of the warp wires presents an interesting example: in 2 places it is to be seen in almost its original thickness and right next to these points, it is almost completely eaten away.

A series of experiments were then carried out:

First Experiment: A piece of mesh was annealed in a hydrogen current for 1 hour at a temperature of 800° and then subjected to the nitric acid test. No selective dissolution of warp wires took place. The loss in weight amounted to 50%, which is therefore much greater than in the case of untreated normal mesh. This mesh is shown in Figure 3 after the nitric acid test. Although no wire was dissolved out, nevertheless the mesh was definitely attacked. Most important, the warp wires were again more strongly attacked than the wove wires. But even here, there were warp wires that were very slightly affected (see a). Besides, it is noteworthy that even the wove wires were much more strongly attacked than they were during the normal test as shown in Figure 2.

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Second Experiment: A piece of mesh was annealed in air at a temperature of 400° for 1 hour and, when subjected to the nitric acid test, no selective dissolution of individual warp wires occurred. The weight loss amounted to only 2%. The microscopic photograph showed one warp wire had undergone a strong attack ("decrease" The rest of the wires remained equally thick. (Figure 4)

Third Experiment: The untreated regular mesh was placed in the solution horizontally, but the susceptible (sic) warp wires were not completely dissolved. The weight loss was 25% and therefore less than when the mesh was placed vertically in the nitric acid.

Fourth Experiment: The mesh was degreased with an ammonia solution and a hand brush. When subjected to the nitric acid test the same condition resulted as in the case of the untreated mesh, that is, dissolving out of individual warp wires.

Fifth Experiment: A piece of mesh was greased with some rape-seed oil in such manner that the rape-seed oil was dissolved in turpentine substitute ("Testbenzin"). The mesh was submerged in this solution and the turpentine substitute was removed from the mesh by volatilization. In this way, a thin film of rape-seed oil would cling to the mesh. The nitric acid test showed the same condition as in the case of untreated mesh, that is, selective dissolving out of individual warp wires.

Sixth Experiment: A piece of mesh was submerged only partially in nitric acid. A microphoto was made of the point of transition ("Übergangsstelle"), which (photo) is reproduced in two different sizes in Figures 5a and 5b. When measuring by means of sliding calipers with Vernier, it was possible to determine that the more strongly attacked warp wires in general were weaker and less round than the warp or wool wires which were not so strongly attacked. These diameters are shown in millimeters in Figure 5 without consideration as to magnification. The red figures refer to the more strongly attacked warp wires; the black figures to the warp or wool wires not so strongly attacked. In Figure 5b the same measurements were carried out on greater photographic magnification. From these measurements it turns out further that the warp wires are as a matter of course weaker as compared with the wool wires. This is understandable when one considers the fact that a greater diameter is required for the wool wires than for the warp wires even in the condition in which they are originally furnished. From these facts emerges a clarification of what is observed: that, in the case of wool wires, a wire is never selectively dissolved out.

Seventh Experiment: A piece of mesh was again partially dipped into the nitric acid. The wires in this case which were selectively dissolved out were carefully removed from the untreated mesh. These wires which were selectively dissolved in the mesh did not show complete dissolution upon individual testing in nitric acid during the above-described period of time.

Microscopic investigation of the surface and structure of a selectively dissoluble and a less dissoluble mesh wire showed no differences. Figures 6, 7, 8, and 9 show such structural (and texture) photographs. Wires were taken from 10 spools of the same batch ("cast") and 10 spools from different batches (pourings or "castings"; R) and drawn out 5, 10, and 15%. Thereupon they were bound together and subjected to the nitric acid test. Within the above-prescribed time no dissolution whatever of individual wires took place.

From these experiments important conclusions can be drawn:

1. Weak oxidation results in a protective layer which reduces the speed of

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- dissolution. It is possible that the wires show the same protective layer or layers of varying thicknesses (third experiment). By annealing in a hydrogen current this protective layer is reduced and thus removed. Other surface layers too, such as oil, rust, and the like are in this way removed so that a sharper attack takes place (first experiment). Of course the other wires are now more strongly attacked so that a selective dissolution of individual wires is not discernible, at least not without a microscopic inspection.
2. It appears, to be sure, that an oil film does not provide a specially protective effect (fourth and fifth experiments).
 3. On the other hand, the wire diameter undoubtedly plays a big part. Thinner wires are dissolved faster than the thicker wires next to them (sixth experiment). This phenomenon is attributable not only to the fact that a thinner (weaker) wire is naturally consumed faster than a thicker one. It is also to be found in the fact that the curvature of a thinner wire is sharper. As is known, a stronger attack occurs as a result of the change in the surface tension of such curvatures as is quite obviously the case, for example, with sheet metal edges and sheet metal corners.
 4. The most important conclusion is that auto-catalysis plays the biggest role in dissolution and in the variable dissolution of individual wires. To this is also attributable the following: the dissolution of metals, among them nickel in nitric acid, takes place in such a way that the metals are first converted into oxides by the oxidizing effect of the nitric acid, which (oxides) are then dissolved in the acid to nitrates. During this dissolving process, brown fumes are formed, which consist of a mixture of nitrogen dioxide and nitrogen oxide (Stickstoffoxyde). These fumes are sometimes called Stickoxyde for short. The Stickoxyde accelerate the oxidation process and consequently the dissolution of the metal. On this account, the observation is made that when the metals dissolve in nitric acid, this takes place slowly at first, but gradually becomes more rapid and finally proceeds furiously. Such a process is called auto-catalysis. During the reaction a material is formed which reacts faster than one of materials used at the outset. The dissolving capability of nitric acid vis-à-vis nickel thus depends to a great extent on the Stickoxyde content or its formation during the testing process. It may well be that nitric acid which has been exposed to the sunlight for some time already contains a certain amount of Stickoxyde as compared with fresh nitric acid, which contains scarcely any. The former then attacks more readily than the latter.

However, other materials, too, can hasten this dissolution capability of nitric acid. Thus one finds that the speed of dissolution of nickel in 40% nitric acid at 30° is retarded by an addition of ferric nitrate (but) accelerated by ferric chloride, iron sulfate, nickel nitrate, and potassium permanganate. (The Corrosion of Metallic Materials, Vol. 2, p. 737). Now, however, nickel nitrate is formed when nickel is dissolved in nitric acid. Thus, this reaction product hastens the dissolution process also and acts auto-catalytically as do Stickoxyde. The distribution of these auto-catalysts is extraordinarily variable in the solution. The Stickoxyd emerges ultimately as a gas and becomes enriched in the upper portion of the solution, whereas the nickel nitrate probably sinks to the bottom as a result of its higher specific gravity.

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Experiments 7 and 8 give the most striking proof of the extraordinary significance of auto-catalysis. For instance, if one subjects only a few wires to the test, no premature dissolution takes place (within the space of 3 minutes). The surface attacked is so small that only a very weak formation of Stickoxyde and nickel nitrate - the two auto-catalysts - takes place so that the speed of dissolution is essentially less than it is if one subjects a piece of mesh to nitric acid - a piece of mesh, say, weighing 2 g with an area of approximately 18,000 mm² and from which some 0.6 g nickel is dissolved with simultaneous formation of Stickoxyde. Another proof is the third experiment where mesh inserted horizontally undergoes a smaller loss in weight and no selective dissolution of individual wires takes place. In this case, the Stickoxyde rise and no longer come in contact with nickel. The nickel nitrate sinks to the bottom and can no longer act auto-catalytically as in the case of the mesh inserted vertically.

In short, it must be determined whether the selective dissolution of individual wires when subjected to the nitric acid test is the result of diameter variations within the limits of the permitted tolerances, of course. In this connection, the presence or absence of thin protective layers of oxide probably also plays a part.

The investigations undertaken show unequivocally that the rapid method chosen, using nitric acid in a dissolving experiment, is wholly unsuitable, since it must lead to extraordinary variations in the results or to false conclusions through the process of auto-catalysis. It has been shown repeatedly, however, that it is misleading, say, to make a preliminary test of individual wires for a faster dissolution co-efficient ("capability") using nitric acid. The nitric acid test must be rejected as an unsatisfactory ("unsuitable for this purpose") rapid test procedure.

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1. [REDACTED] Comment. The supporting material for the above report is available in OSI/NED.

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